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Internal sulfate attack in mortars containing contaminated fine recycled concrete aggregates

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HIGHLIGHTS

- Gypsum residues in fine recycled aggregates can cause internal sulfate attack.
- Limited porosity or thaumasite formation had an aggravating role on the swelling.
- Increased alkalinity or limited sulfate contents inhibited the swelling response.

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ABSTRACT

Internal sulfate attack can be caused by the gypsum residues present in fine recycled aggregates (FRA). As opposed to the better known external sulfate attack or Delayed Ettringite Formation (DEF), the sulfates in this context are provided by a gypsum contamination of the aggregates. Mortars made with contaminated FRA were subjected to different conditions, to assess which parameters had an influence on the sulfate attack reaction. Their mechanical properties and microstructure are investigated. Results showed that gypsum content, porosity, temperature and alkalinity influenced the consequences of sulfate attack. However, the gypsum size distribution and cement type did not.

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1. Introduction

1.1. Gypsum in fine recycled concrete aggregates

One of the key points within the framework of a sustainable construction sector is the recycling of its waste products, completing the life cycle of these materials. Recycled concrete aggregates (RCA), obtained by the demolition or deconstruction of older concrete structures, can be used in a new concrete as a replacement for natural aggregates [1]. 1.7 tonnes of these RCA are produced per person per year in Europe, waiting to be valorized [2]. Gypsum ($CaSO_4.2H_2O$) is used in the construction sector firstly as an addition to Portland cement, to regulate the setting time of concrete and prevent a flash set [3]. Besides that, gypsum is the major constituent of plaster walls in buildings. RCA, as a consequence, will contain a certain amount of gypsum. Larger concrete and gypsum particles can be separated from each other based on a difference in color [4] or density [5], but these techniques are not applicable on the smallest size fractions of RCA. In fine recycled aggregates (FRA), gypsum is an important contaminant to be considered: the water soluble sulfates coming from the gypsum particles strongly limit their valorization potential [6].

1.2. Sulfate attack: sources and mechanisms

Sulfate attack is a deteriorating process where sulfates react with water and aluminate hydrates in a hardened cement paste to form secondary ettringite. It is assumed that this mineral exerts a pressure on its surrounding cement paste and causes a volumetric deformation [7]. Macroscopically, the concrete structure will show swelling behavior and the formation of cracks. Ettringite is a normal hydration product in the cement paste: its formation only becomes dangerous when it occurs after setting, in a rigid cement matrix. Depending on the source of the sulfates responsible for the reaction, a distinction can be made between an external and an

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internal reaction. To experience *external* sulfate attack, the structure is submerged in a sulfate rich environment such as soil or seawater. Diffusion mechanics and microcrack propagation from the surface inwards are determining factors here [8]. *Internal* sulfate attack happens when there is a delayed release of sulfates from the hardened cement matrix. In this sense, an internal source of sulfates eliminates the diffusion and microcrack necessity of external sulfate attack, possibly accelerating the reaction. Delayed Ettringite Formation (DEF), which occurs when high curing temperatures have destroyed the sulfate hydrates that were initially formed [7], has been known for some time. The current situation, where FRA is contaminated with gypsum residues, is a relatively new problem that has not been studied extensively.

The following aspects of the internal sulfate attack reaction were selected to be elaborated in this study:

Alkalinity Many authors show the important role of the alkalinity of the interstitial solution, as it interferes with the equilibrium between the different sulfate phases. A higher alkalinity favors the existence of monosulfate and the absorption of sulfur on the C-S-H gel instead of the formation of ettringite [9], so ettringite formation triggers as pH lowers. Nevertheless, a higher swelling due to ettringite formation is often found in mixes with a higher alkalinity [9–13]. While alkalinity speeds up hydration and increases early compressive strength [14], it leads to lower mechanical performances in a sulfate presence [9,10]. Besides a possible interaction with sulfate attack, alkalinity is also a risk factor for the alkali-silica reaction and other durability issues.

Cement type Using a sulfate-resisting cement allows the use of FRA with a high sulfate content [15]. These types of cement contain less C_3A, one of the reactants needed to form ettringite. Moreover, fewer gypsum is added to this cement type as a setting retarder, compensating for the additional sulfate source to which the mixture will be exposed. The SO_3/Al_2O_3 ratio of a cement is an important factor regarding its potential to form ettringite [16].

Porosity The most commonly accepted theory about the cause of expansion is the heterogeneous crystal pressure exerted by the growing ettringite crystals [7,3,17]. In this sense, a lower porosity means more confinement and a higher internal pressure. On the other hand, in the case of external sulfate attack, a lower porosity would prevent the inwards diffusion of sulfates and thus limit the swelling potential [18].

Sulfates Before dissolution, sulfates can be associated with different cations. It has been found that the sulfates originating from Na_2SO_4 lead to more swelling than those from $CaSO_4$ [9], and that $MgSO_4$ is even more damaging [19]. This would suggest that a gypsum contamination is less damaging than other types of sulfate attack that have been researched. However, these are external sources. The rapid availability of an internal $CaSO_4$ contamination could make this difference smaller.

Coarser gypsum particles are hypothesized to not feed early ettringite formation, but react later in an already rigid cement matrix. To keep the risk on sulfate attack at a reasonable level, the current water soluble sulfate limit in coarse recycled aggregates is established at 0.2% by EN 206 [20], with no mention of FRA. At these quantities, sulfates are considered the limiting reagent in the ettringite formation reaction so any augmentation would hypothetically lead to more swelling. The conclusions of recent durability studies indicate a level of 0.3% should be made possible [21].

Thaumasite formation Next to ettringite, sulfates can also contribute to the formation of the expansive mineral thaumasite. While damage caused by thaumasite is more severe than that caused by ettringite, thaumasite formation does not occur as often [22]. Only at temperatures lower than 10 °C and in the presence of a carbonate source, is thaumasite favored over ettringite [23].

1.3. Objectives

In this study, contaminated FRA were used in mortars to research the damaging effects of sulfates. Each parameter of interest was varied while others were kept constant, to identify the factors that can worsen or mitigate the sulfate attack results. Knowing which parameters to manipulate in a mix design with highly contaminated FRA will ultimately promote the use of these recycled aggregates. The results of this study could also contribute to the ongoing discussion about the sulfate attack reaction mechanism [24].

2. Materials and methods

2.1. Used materials

FRA were made in the laboratory by fabricating a concrete and subsequently crushing it. The composition of this original concrete is given in Table 1, and was designed to obtain a consistency class S3 and strength class C30/37. After 90 days of curing, this concrete was crushed by a jaw crusher and the resulting 0/4 mm fraction was used as FRA in all described tests. The use of this 'model' FRA gave exact control of the chemical composition of the materials and removed any possible variability or contamination at the level of the aggregates by chlorides, organics, etc. This FRA was then manually contaminated with gypsum to obtain a 'clean' material where only sulfates could contribute to a deteriorating reaction.

Fig. 1 and Table 2 summarize the properties of the resulting FRA. Water absorption and particle density of the FRA were determined via the method described by Zhao et al. [25]. Characterization techniques for natural aggregates, described in EN 1097-6 [26], consistently underestimate the water absorption of FRA because of the fineness and agglomeration issues between the particles. The method – designed in response to this difficulty – by IFSTTAR [27] seems to overestimate the water absorption of FRA but works well for particles in the 0.5/4 mm range. Thanks to an very good correlation between the hardened cement paste content or mass loss at 475 °C and the water absorption, the water absorption of the fines can then be extrapolated. Using the water absorption of each size fraction (either measured for the coarser particles or calculated for the fines) is more accurate than using either of the two mentioned experimental methods for the whole 0/4 mm bulk [28]. Even though no gypsum was added to the model concrete, 0.18% of water soluble sulfates were measured via ion chromatography, originating from the used cement.

The gypsum used to contaminate this FRA was a $CaSO_4.2H_2O$ powder (D50 13 μm) obtained from VWR Chemicals. The sulfates from this gypsum contamination are added to the 0.18% of water soluble sulfates already in this FRA, originating from cement particles. A CEM I 52.5 N cement from HOLCIM was used as the default cement, in one test replaced by a High Sulfate Resisting (HSR) CEM I from the same manufacturer. The chemical composition of these cements is shown in Table 3.

2.2. Mortar fabrication

All mortars were prepared with the FRA described in Section 2.1, and were contaminated with 5% (by weight of the granular fraction) of gypsum. This gyspum, together with the residual sulfates in the model FRA, brings the total sulfate content of this mix to 3.08%. 7 days before mixing, the FRA was presaturated with its absorbed water and 10% of the mixing water. The standard procedure described in EN 196-1 [29] for mortar fabrication was followed, where normalized sand was replaced volumetrically by

Table 1
Composition, in kg, of the original concrete.

CEM I 52.5 N	Water	Limestone aggregates (mm)				Superplasticizer
		0/4	2/7	7/14	14/20	
350	175	216	658	436	612	0.4%

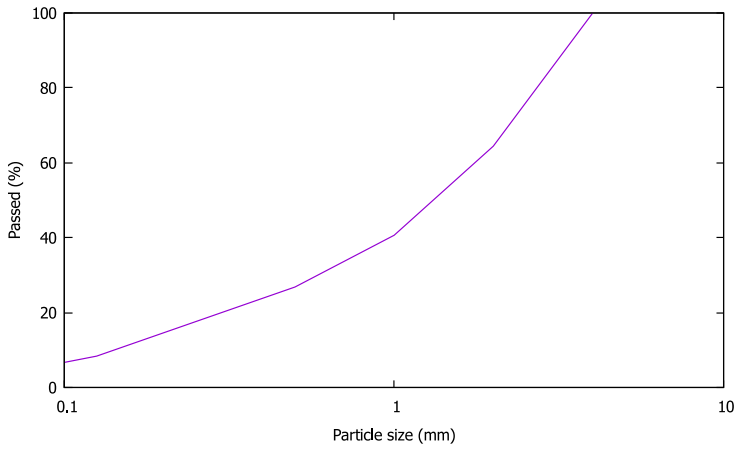

Fig. 1. Size distribution 0–4 mm of the used FRA.

Table 2
Characterization of the used FRA.

Water absorption	Particle density	SO_4^{2-} content
9.78%	1.95 g/cm^3	0.18%

Table 3
Chemical composition (mass%) of the used cement types.

Chemical	CEM I 52.5 N	CEM I HSR
CaO	64.3	64.6
SiO_2	18.3	21.4
Al_2O_3	5.2	3.7
Fe_2O_3	4.0	4.6
MgO	1.4	0.8
Na_2O	0.32	0.27
K_2O	0.43	0.40
SO_3	3.5	2.5
Cl^-	0.06	0.06
LOI	2.3	1.3
C_3A	6.6	2.4
C_4AF	12	14
C_3S	61.9	68.8
C_2S	11.2	9.4

FRA: using a particle density of 2.6 g/cm^3 for normalized sand and 1.95 g/cm^3 for the FRA, the aggregate envelope volume was kept constant. After a cure of 24 h, the mortars were kept in water at 21 °C. These described compositions or conditions were then varied accordingly, depending on the parameter that was tested.

2.3. Tested parameters

The following parameters were hypothesized to have an effect on the internal sulfate attack reaction. The influence of each one was tested with an exaggerated high (+1) and a low (−1) level, and in one case also an intermediate (0) level. Table 4 summarizes these levels and how each of them was obtained by adapting the standard mortar composition. Table 5 shows the compositions in more detail. To isolate the response of only one parameter, each series of replicates is kept in its own container, so as not to be influenced by the leaching water of another [30]. While one factor is being researched, all other parameters are kept as described in Section 2.2.

Table 4
Summary of how the mortar composition was changed to obtain the levels of the different parameters.

Factor	Level	
Alkalinity (A)	−1	0.61% Na_2O Eq.
	+1	1.2% Na_2O Eq.
C_3A content (C)	−1	2.4% (CEM I HSR)
	+1	6.6% (CEM I)
Gypsum grain size (G)	−1	Powder (D50 13 μm)
	+1	2/4 mm distribution
Porosity (P)	−1	W/C 0.35
	0	W/C 0.5
	+1	W/C 0.65
Sulfate content (S)	−1	0.47% of water soluble sulfates
	+1	3.08% of water soluble sulfates
Temperature (T)	−1	5 °C and a carbonate addition
	+1	21 °C

Alkalinity The lower level of this parameter is the normal alkalinity present in the used cement. The higher level is double this amount, achieved by adding NaOH to the mixing water.

Cement type To test the influence of the available C_3A, a HSR cement was used. A small difference in alkalinity between the HSR cement and the CEM I was mitigated by adding NaOH to the mixing water, bringing the Na_2O Eq. of both cements on the same level.

Gypsum grain size The same fine gypsum powder used in the other samples, was hardened and subsequently crushed to obtain particles in the 2/4 mm range. These two size distributions – the powder or the coarser particles – are used to contaminate the FRA.

Porosity To research the influence of the available porosity, the water to cement ratio (W/C) was varied.

Sulfate content A sulfate amount, one order of magnitude smaller than in the other mortars, was used to demonstrate the importance of this parameter.

Table 5
The compositions, in kg, of the different mortar samples. The changes between a mix and its reference composition is placed in bold. For the C-1 samples, the difference is in the *type* of cement, for the G+1 samples the *size* of the gypsum particles. Since the gypsum contamination is expressed as a mass% of the aggregate part, a lower gypsum content in S-1 means more FRA and thus also more absorbed water.

Name	Cement	FRA	Absorbed water	Mixing water	Gypsum	Extra
A-1, C+1, G-1, P0, S+1, T+1	1.35	0.96	0.094	0.675	0.05	
A+1	1.35	0.96	0.094	0.675	0.05	**10.30 g NaOH**
C-1	1.35	0.96	0.094	0.675	0.05	**1.2 g NaOH**
G+1	1.35	0.96	0.094	0.675	0.05	
P-1	1.35	0.96	0.094	**0.473**	0.05	
P+1	1.35	0.96	0.094	**0.878**	0.05	
S-1	1.35	**1.005**	**0.098**	0.675	**0.005**	
T-1	**1.08**	0.96	0.094	0.675	0.05	**270 g limestone filler, 2.1 g NaOH**

Thaumasite formation These mortars were kept in water at 5°C to promote thaumasite formation over ettringite. For thaumasite formation, a source of carbonates is necessary besides C_3A, sulfates and water. 20% by mass of CEM I was therefore replaced with a limestone filler. Here again, NaOH was added so the alkalinity of the mix resembled the others.

2.4. Monitoring of the reaction

To follow the development of the internal sulfate attack reaction, the mortar specimens were subjected to different tests. On a macroscopic level, the mass, length and ultrasonic wavespeed were recorded weekly to observe features of sulfate attack such as swelling and possible internal cracking. At 7, 28, 90 and 180 days the mortars were characterized mechanically for their compressive strength [29] and porosity by Mercury Intrusion Porosimetry. Every described test was done for 3 replicate mortars. A microstructural analysis for the samples at 90 days was carried out to examine the pore structure and ettringite deposits, to complement the swelling results and provide grounds for their interpretation. The samples were prepared according to routine procedures of embedding and polishing [31] using a 2020 resin from Huntsman and MD System from Struers with water-free diamond pastes and lubricants. Images in scanning electron microscopy were obtained on a Hitachi S-4300/SE-N and coupled with EDS analyses.

3. Results and discussion

In Fig. 2, the length change of the mortars is shown. Standard deviations are not shown on these figures to improve their readability, but are taken into account when performing an unpaired t-test to check whether the swelling results differ from each other statistically.

According to Table 6, C-1, G+1 and P+1 are similar to the standard mix where all parameters have their normal value. Four other samples deviate from this trend: P-1 and T-1 had a larger expansion, A+1 and S-1 had a lower expansion. Next to their length, the mechanical properties that were monitored are presented in Fig. 3. The compressive strength of all samples kept steadily increasing over time but did not seem to have any correlation to the corresponding swelling amounts. The samples that showed a high or low swelling did not have a low or high compressive strength, respectively. There was a large variation of the measured strengths between 25 and 40 MPa, the parameters that did not influence the swelling amount did influence the compressive strength. There was less variation between the samples in terms of porosity, only the sample with a limited W/C ratio had a distinct lower porosity. The macroscopic differences between the parameters are interpreted together with microstructural observations.

3.1. General case

The four curves on Fig. 2 that stay together between the 0.06% and 0.08% marks, show that increasing the porosity, using larger

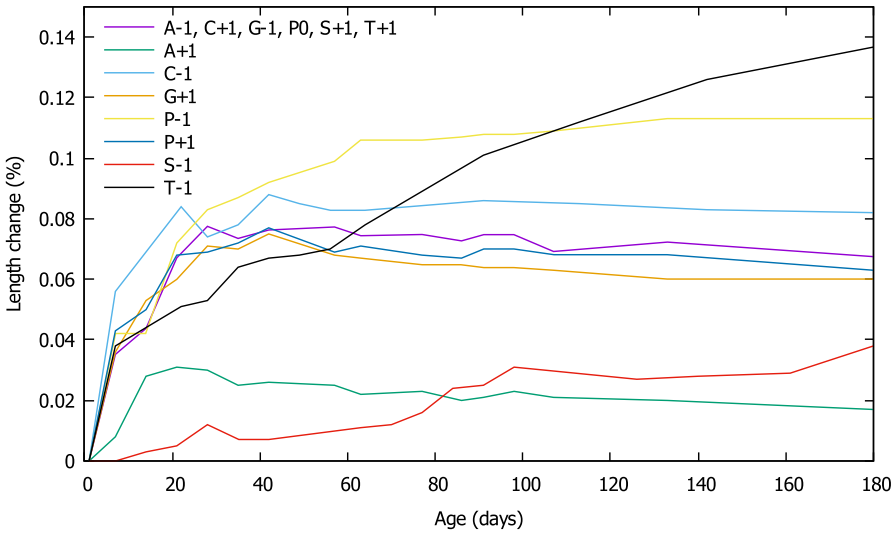

Fig. 2. 6 month swelling behavior of the mortar samples in function of the tested parameters.

Table 6
Unpaired t-test to evaluate if there is a significant difference between the samples and their reference mix, shows that 3 parameters did not change the swelling results (C-1, P+1 and G+1), and 4 did (T-1, P-1, S-1, A+1).

Name	Length (180 days)	Stdev (180 days)	Sample size	Difference for $p > 0.05$
A-1, C+1, G-1, P0, S+1, T+1	0.0675	0.007	3	
A+1	0.017	0.002	3	yes
C-1	0.081	0.005	3	no
G+1	0.060	0.002	3	no
P-1	0.113	0.004	3	yes
P+1	0.063	0.004	3	no
S-1	0.037	0.010	3	yes
T-1	0.137	0.004	3	yes

gypsum grains, or limiting the available C_3A did not influence the amount of swelling. SEM results confirmed that the morphology of these samples was very similar. Fig. 4 is a typical image found with recycled materials: the recycled aggregate is a cluster of natural aggregates in the original cement paste. The new cement paste, surrounding the recycled aggregates, had a notably higher air content, which has been observed before by Bouarroudj et al. [32] for the same material. This air content can be explained by the surface roughness of the recycled aggregates [33], which captures more air into the mixture than a round (natural) aggregate. Next to that, the difference in surface free energy between the new cement paste and the recycled aggregates could also have played a role in this elevated air content [34]. Fig. 5 illustrates how ettringite deposits in these samples were mainly found in pores or air bubbles, which sometimes also resulted in cracks in the surrounding paste.

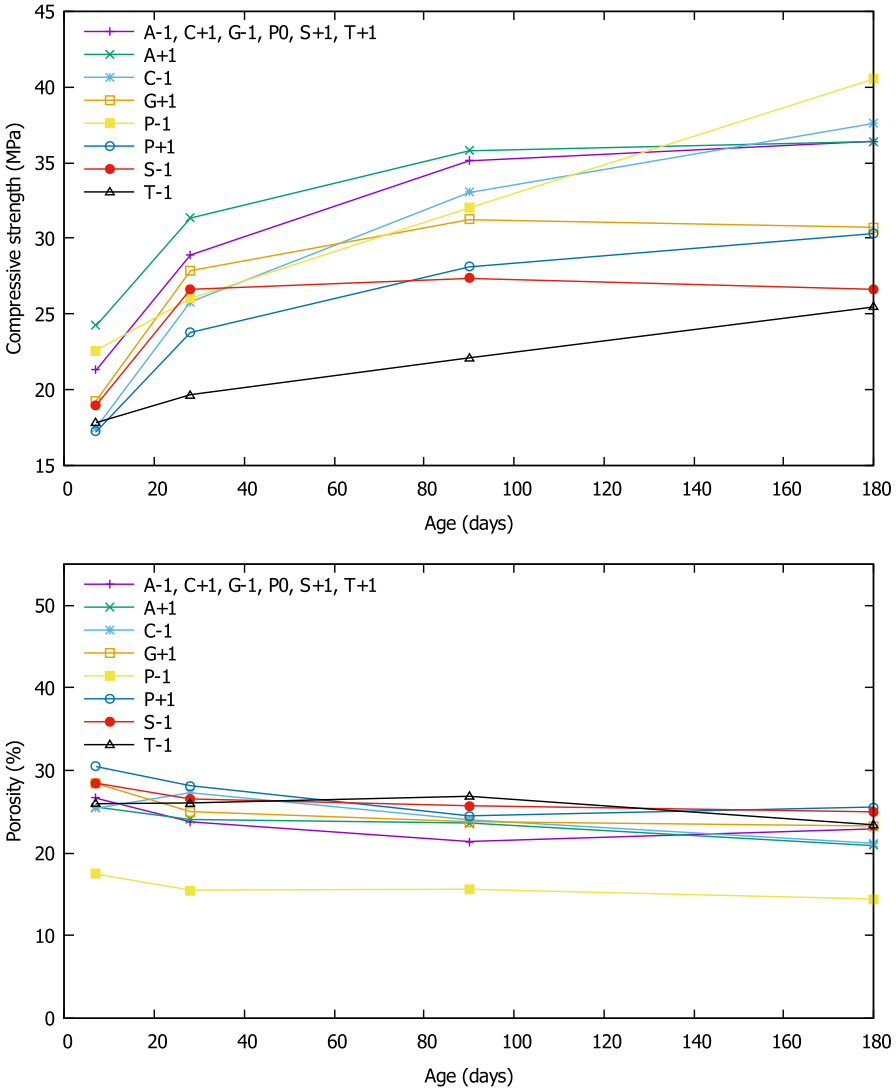

Fig. 3. Compressive strength and porosity of the samples at 7, 28, 90 and 180 days.

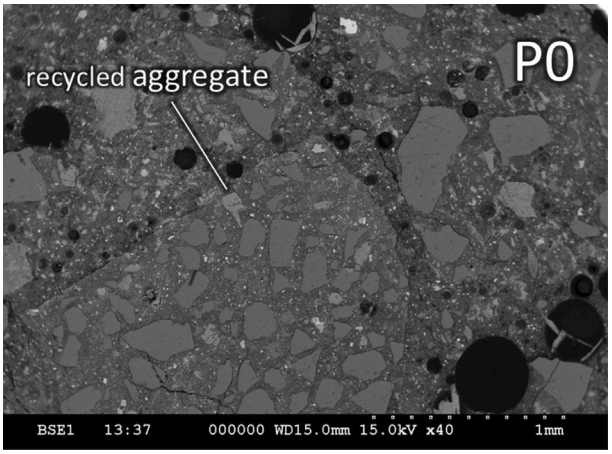

Fig. 4. Contrast between a recycled aggregate and the new cement paste with a high air content.

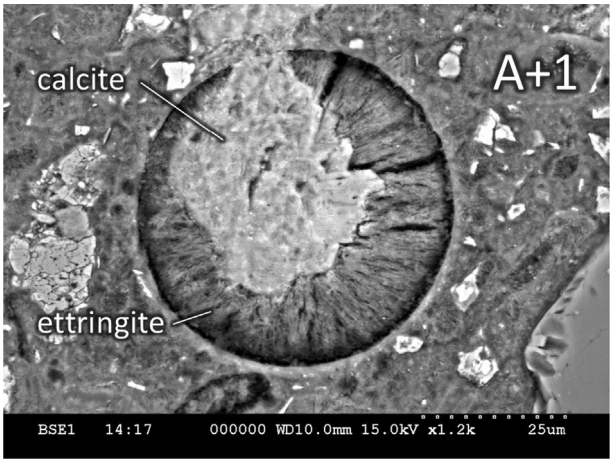

Fig. 6. A piece of calcite promoted the formation of ettringite at high alkalinity at an early age.

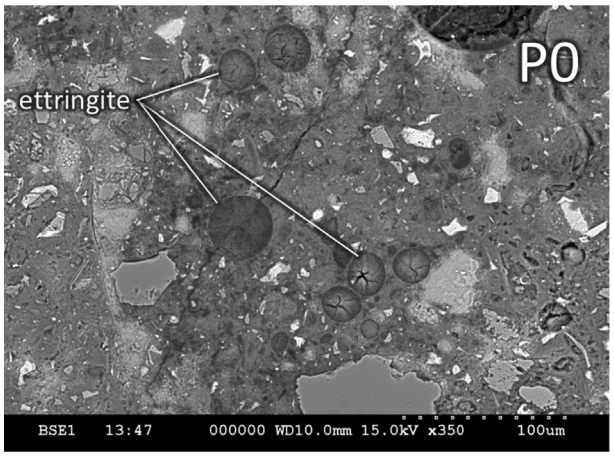

Fig. 5. Ettringite was found in pores and air bubbles, which exerted a pressure and cracked the surrounding cement paste.

3.2. Alkalinity

Sample A+1 with an increased alkalinity did not show any evidence of cracks. Air bubbles were often filled with ettringite, but not in a way that caused damage. Instead, a high percentage of them showed the presence of a piece of calcite or portlandite in their center, with ettringite crystals growing outwards of this center and not inwards from the cement paste. This is demonstrated in Fig. 6. Calcite has been described as a nucleation center for very fast ettringite growth at high alkalinity [35,36]. The kinetics of this reaction explain the lack of available sulfates to cause damage in a cured cement matrix. A higher alkalinity has also been observed by Juenger et al. [14] to increase the initial rate of hydration and cause a higher early compressive strength, which has been confirmed in Fig. 3. However, the swelling and compressive strength results obtained in these experiments contradicted the findings of numerous authors described in Section 1.2. This could be due to the chosen experimental setup: the high alkali values in this study were obtained by adding NaOH, which is immediately available. Alkali's coming from the adherent cement paste of FRA would take more time to leach into the interstitial solution. The rapid initial hydration in this sample prevented the higher swelling that is normally provoked by the alkalinity of FRA.

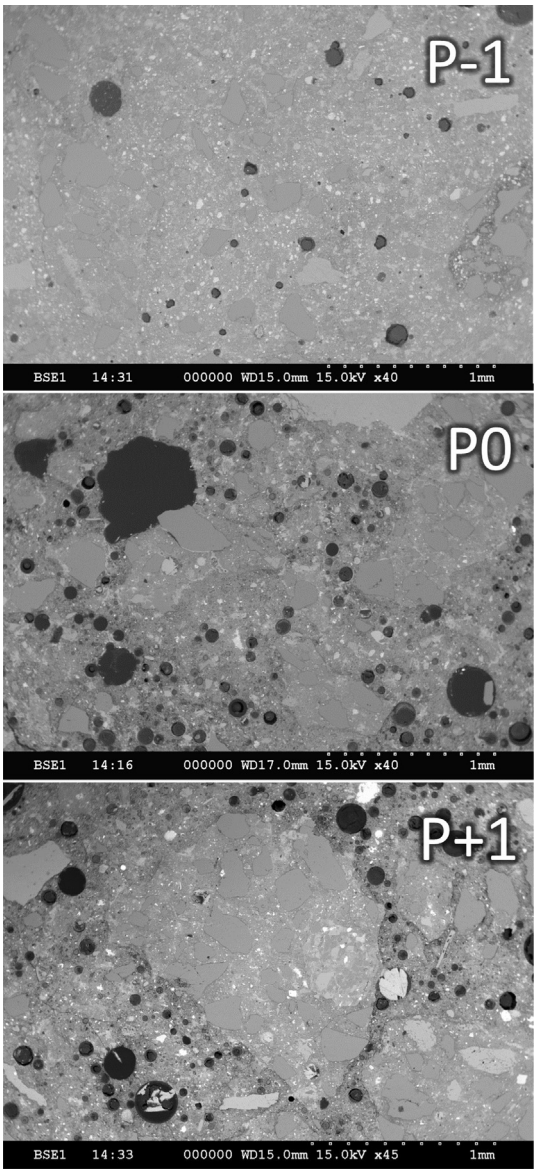

Fig. 7. Comparison of the cement paste density between P-1, P0, and P+1.

3.3. Porosity

While increasing the W/C ratio did not have an effect on the reaction, lowering it raised the swelling amount. Fig. 3 shows a distinctly lower porosity for sample P-1, but not an equally large difference between P0 and P+1. In Fig. 7, these findings are confirmed by showing a similar morphology for P0 and P+1, but a significantly denser matrix for P-1. No superplasticizer was added to counteract a loss in workability due to the lower W/C. The microstructure of P-1 also showed the presence of more unreacted cement particles. The P-1 samples displayed many cracks throughout which were filled with ettringite crystals, as illustrated in Fig. 8. A high swelling level would seemingly be a good indicator for internal damage.

3.4. Sulfates

The S-1 samples contained only 0.28% of water soluble sulfates, which is still well above the maximum allowable limit in recycled aggregates [20]. Still, no significant damage occured, as shown by its swelling behavior in Fig. 2 and microstructure in Fig. 9. The long term compressive strength of these samples, shown in Fig. 3, was lower than those with higher sulfate contaminations, but only because it did not increase after the first month of aging.

3.5. Temperature

The samples kept at lower temperatures showed an important amount of swelling compared to the other mixes, but the reaction seems slower and did not reach a stabilization point yet after 6 months. Compressive strength, shown in Fig. 3, was much lower compared to the other samples, which is due to the limited amount of cement in this mix. Fig. 10 shows the internal damage in these samples, while EDS confirmed that pores, air bubbles or cracks are filled with an ettringite/thaumasite mixture.

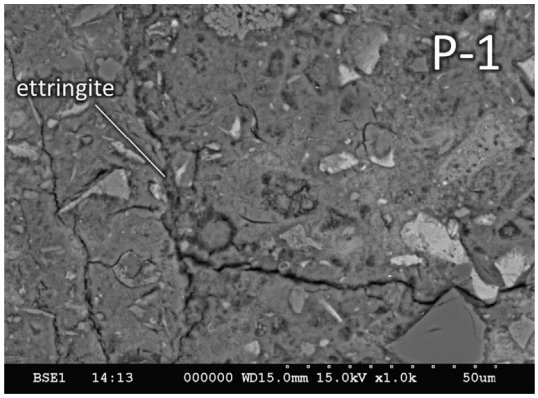

Fig. 8. Cracks filled with ettringite in a sample with lower porosity.

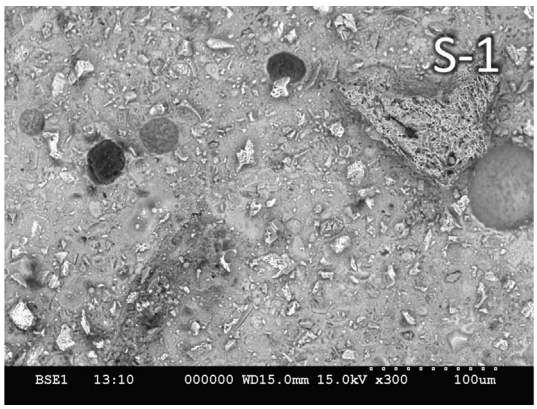

Fig. 9. Lowering the sulfate content resulted in an undamaged sample.

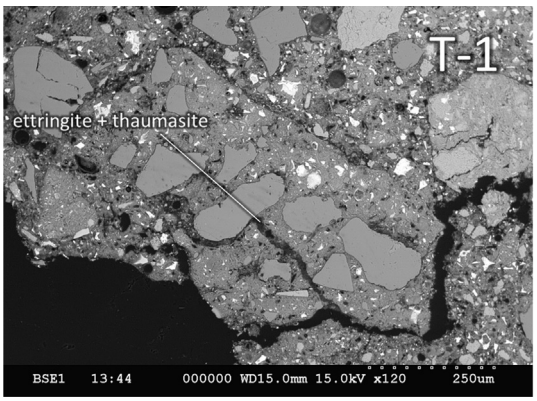

Fig. 10. The T-1 samples were damaged by a mixture of ettringite and thaumasite.

4. Conclusion

The water soluble sulfates in FRA are responsible for a deterioration when they react with C_3A and water in a new cementitious mix. These sulfates could originate from gypsum residues at the demolition site, but also from cement particles in otherwise 'uncontaminated' FRA. The severity of this deteriorating reaction is determined by other factors: some can limit the swelling potential, others enhance it. Six parameters were chosen to research and their influence on the sulfate attack reaction has been identified. The use of a model FRA manually contaminated with gypsum made it possible to isolate the responses of only one parameter at a time, without interference of other variabilities or contaminations.

Limiting the C_3A content, using coarse gypsum particles, or augmenting the W/C ratio did not significantly change the swelling results. The formation of thaumasite or limiting the available porosity lead to a larger expansion. On the other hand, increasing the alkalinity of the mix or using a lower sulfate content seemed to limit the swelling results. This indicates that the maximum sulfate content established in EN206 may be too strict, which is in line with the findings of the PN RecyBéton [21] and their subsequent proposal to set this limit at 0.3%. Unrelated to their influence on the swelling amount, the variation of these parameters also changed the compressive strength of the mortars.

In general, the use of FRA in mortars led to a high air content, which was responsible for a lower compressive strength when compared to the compressive strength of a standard mortar with natural aggregates. This air content was explained by the surface roughness of recycled aggregates.

These results provide industrials with helpful information for mix designs with high sulfate contents. This could in turn help promote the use of contaminated FRA, which are up to now not valorized.

Future work should focus on upscaling these tests to concrete, and/or research a possible interaction between the parameters.

CRediT authorship contribution statement

C. Colman: Investigation, Writing - original draft, Visualization. **D. Bulteel:** Supervision, Writing - review & editing. **V. Thiery:** Writing - review & editing. **S. Rémond:** Investigation, Writing -

review & editing. **F. Michel:** Writing - review & editing. **L. Courard:** Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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